PATENT SPECIFICATION

763,383

Date of Application and filing Complete Specification: June 25, 1954.

No. 18772/54.

Application made in Germany on July 11, 1953.

Complete Specification Published: Dec. 12, 1956.

Index at acceptance:—Classes 81(1), E1A(4A2:4A3:7B:14A:14C:16:17); and 91, D2(B:E:F:G:K:L:N:P:Q:W), S2(B:E:F:G:K:L:N:P:Q:W).

COMPLETE SPECIFICATION

New Bromine-containing Washing and Rinsing Agents

I, HEINZ WALLRATH, of 65a, Heidter Berg, Wuppertal-Barmen, Germany, a German Citizen, trading as DR.MED. JOSEF ELLENDORFF & Co., do hereby declare the invention, for which I pray that a patent may be granted to me, and the method by which it is to be performed, to be particularly described in and by the following statement:—

The invention relates to disinfecting washing and rinsing agents containing active bromine.

The use of inorganic or organic chlorine compounds in washing agents for disinfecting purposes is known. Additions of considerable quantities of substances liberating active chlorine are necessary however for effective disinfection.

Specification No. 392,814 discusses previously proposed additions of alkali salts of aryl-sulpho-N-halogen amides, for example sodium para-toluene sulpho-chloramide, to hypochlorite solutions, and states that a disadvantage of such disinfectant detergents is the diminution in the disinfectant action, particularly in contact with organic liquids. It is proposed in the said Specification to avoid this disadvantage by the use of mixtures of hypochlorites with alkali salts of aryl-sulpho-N-halogen amides, for example alkali salts of para-toluene sulpho-chloramide, either as the aqueous solutions as such or as mixtures of such solutions with alkaline substances employed as detergents.

In the book "Arzneimittel-Synthese" by H. P. Kaufmann (Springer-Verlag 1953) it is stated on page 522 that bromine has a weaker bactericidal action than chlorine, and that the synthesis of compounds which split off bromine, for example a bromine compound corresponding to chloramine, is therefore without interest.

It has now surprisingly been found that in a certain compound namely sodium *p*-toluenesulphonbromamide, bromine has a better bactericidal activity than chlorine, and accordingly the present invention provides a disinfectant washing or rinsing agent containing sodium *p*-toluenesulphonbromamide and a detergent. The bromine in sodium *p*-toluenesulphonbromamide is made available at a more favourable rate than the chlorine in the corresponding chlorine compound.

For brevity, the compound sodium *p*-toluenesulphonbromamide will be referred to herein as "bromamine": this compound is practically odourless and can readily be prepared pure, and can be used with excellent results for the production of odourless or perfumed disinfectant washing agents, even in small proportions which are far less than the proportions of bromine compounds hitherto for purposes of disinfection, alone or together with other disinfectants, because its content of active bromine is very high, being found by iodometric titration, for example, to be equal to the bromine content of the compound.

The bromine effect obtained with bromamine is due to the fact that bromamine reacts with water to form hypobromite by the known equation:

$$R.SO_2N\begin{matrix}Na\\Br\end{matrix} + H_2O \longrightarrow R.SO_2NH_2 + NaOBr$$

where R is the toluene residue: $CH_3.C_6H_4$—.

Preferably the bromamine content of the disinfectant washing or rinsing agents is 0.05 to 5% by weight of the final composition.

Unlike nuclear bromine substituted compounds, with which this reaction occurs either not at all or very slowly, bromamine behaves as a salt in which the NaBr is relatively loosely bound and reacts with water, in acid, neutral or alkaline medium, to yield hypobromite, whereas in the washing agent itself it remains firmly bound to the amide residue.

Thus the bromamine-containing washing and rinsing agents of the invention represent the discovery of new stable cleansing agents

which preserve laundry, are non-poisonous and practically odourless, so that laundry treated therewith retains the smell of fresh laundry after washing without exhibiting the usual smell of sterilising agent or disinfectant.

This disinfection with simultaneous cleaning, which is of importance principally as a prophylactic measure, but also during epidemics and in the case of infectious diseases, is obtained simply by normal ordinary washing with the new washing agents. Antiseptic and prophylactic cleansing of the body can also be effected by washing with the washing agents of the invention. The step of adding bromamine to washing agents is novel and represents a substantial technical and hygienic advance in the development of disinfectant cleansing agents.

The washing agents with the desired bromine effect, which are practically odourless and can consequently be readily and permanently perfumed, can also be used with the same effect for cleaning and disinfecting floors, furniture and the like in hospitals, sanatoria, sick-rooms and so on, their pleasant fresh smell being appreciated as particularly advantageous.

Fatty acid salts and also surface-active synthetic foaming and wetting agents of all kinds can be used as detergents and components of the rinsing agents.

The usual additions, such for example as per-salts, sodium carbonate, sodium bicarbonate, Glauber's salts, sodium chloride, soluble phosphates of all kinds, silicates, cellulose ethers, cellulose esters, cellulose glycollates, starch, perfumes, dye-stuffs, superfatting agents, abrasives and sterilisers can be incorporated in the washing and rinsing agents without reducing the bromine disinfecting action. Further disinfectants can also be added in a proportion of 0.01 to 0.5% by weight of the final composition.

The washing and rinsing agents of the invention can be employed as stable powders for cleaning laundry, floors and the like or as shaped pieces of tablets for cleansing the body or in the form of pastes (for example soft soaps) and solutions.

The invention is illustrated by the following examples but is not limited by these examples; on the contrary the bromamine can be added to any commercial detergent-containing washing and rinsing agent to produce stable and storable antiseptic cleansing agents:

EXAMPLE 1. (Soap powder).

Alkali salts of fatty acids obtained by saponification of a mixture of:	
80 parts tallow	
15 parts coconut oil	
5 parts olive oil	35.00%
Sodium carbonate	33.00%
Waterglass	7.50%
Calcite	1.00%
Magnesium sulphate	1.25%
Sodium bicarbonate	1.75%
Sodium perborate	4.00%
Perfume and dyestuff	0.50%
Water	15.50%
Bromamine	0.50%
	100.00%

EXAMPLE 2. (Washing powder)

Detergent mixture of the sodium salts of a C_8—C_{18} fatty alcohol sulphate ($\frac{1}{3}$) and of dodecyl benzene sulphonate ($\frac{2}{3}$)	12.50%
Sodium carbonate	44.00%
Waterglass	7.50%
Sodium cellulose glycollate	2.00%
Water	32.475%
Calcite	1.00%
Bromamine	0.50%
Halogenated 2.2¹-dihydroxy-diphenyl methane	0.025%
	100.00%

EXAMPLE 3. (Toilet soap)

0.50% of bromamine is added to commercial toilet soap.

EXAMPLE 4. (Fine washing agent).

Sodium Lauryl	35.00%
Sodium polyphosphate	10.00%
Sodium bicarbonate	5.00%
Perfume mixture	3.00%
Anhydrous sodium sulphate	46.00%
Halogenated phenol	0.50%
Bromamine	0.50%
	100.00%

To determine the disinfectant action, Sabouraud nutrient solutions are inoculated on the one hand with red yeast and on the other hand with epidermophytes (Kaufmann-Wolf) and a type of endomyces and staphylococcus; in each case 0.5, 1 or 2 cc. of washing solution, which contains the soap powder

or washing powder in a concentration of 1:100 and a bromamine addition, for example of 0.46%, calculated on the soap powder or washing powder, is mixed with 15 cc. of nutrient solution. A minus (−) indicates that all the bacteria are killed; a plus (+) indicates that a culture grew but rapidly died; two plus signs (++) indicate that the bacteria survive. The temperature of the action is 25°C.

There are used on the one hand a commercial washing powder containing 12% of detergent (one-third alkyl sulphonate and two-thirds alkyl benzene sulphonate), 44% of sodium carbonate and the usual additions (the bromamine addition is 0.46%), and on the other hand a commercial self-acting soap powder with 35% of fatty acid, 33% of sodium carbonate and 4% of perborate together with the usual additions (in this case also the addition of bromamine is 0.46%).

TABLE 1

Substance	Proportion of disinfecting addition in the powder	Concentration of washing powder in the experimental solution	Concentration of the disinfectant addition in the experimental solution	Cultures introduced	Cultures which grew (++)	Cultures which grew but rapidly died (+)	Cultures which did not grow (—)
Blank	—	—	—	6	6	0	0
Washing powder	—	1:850 (2 cc. washing solution + 15 cc. nutrient solution)	—	6	4	2	0
Washing powder with bromamine	0.46%	1:850 (2 cc. washing solution 15 cc.+ nutrient solution)	1:185,000	6	0	1	5
„	0.46%	1:1600 (1 cc. washing solution + 15 cc. nutrient solution)	1:347,826	6	2	0	4
Soap powder	—	1:850 (2 cc. soap solution + 15 cc. nutrient solution)	—	6	4	2	0
Soap powder and bromamine	0.46%	1:3100 (0.5 cc. soap solution + 15 cc. nutrient solution)	1:673,913	6	2	1	3
„	0.46%	1:1600 (1.0 cc. soap solution + 15 cc. nutrient solution)	1:347,826	6	0	1	5

TABLE 2

DISINFECTION TEST BY A SUSPENSION EXPERIMENT

0.5 Staphylococci suspension of three fresh stocks in 4.5 cc. solution

Solution	%	Period of action: 2 mins.	10 mins.	30 mins.
41 b	5%	—	—	—
41 b	1%	—	—	—
41 b	0.5%	++++	++	—
42 b	5%	—	—	—
42 b	1%	—	—	—
42 b	0.5%	++++	+	—
Control with commercial soap	5%	—	—	—
	1%	++++	+++	++
	0.5%	++++	++++	++++
Control without soap		++++	++++	++++

0.5 B. Coli suspension of three fresh stocks in 4 5 cc. solution

41 b	5%	—	—	—
41 b	1%	—	—	—
42 b	5%	—	—	—
42 b	1%	—	—	—
Control with commercial soap	5%	—	—	—
	1%	+++	—	—
	0.5%	++++	++++	—
Control without soap		++++	++++	++++

NOTES ON TABLE 2.

No. 41*b* is a 1% solution of a commercial soap powder with a content of 0.23% bromamine.

No. 42*b* is a 1% solution of the same soap powder with a content of 0.46% bromamine.

The control is made with commercial soap with a content of 2% of halogenated dihydroxydiphenyl methane in 1% solution. 0.5 cc. of the suspension is then treated with 5%, 1% and 0.5% of these 1% solutions and made up to 4.5 cc.

A minus (−) indicates that everything has been killed. Plus signs (+, ++, +++, ++++) show surviving bacteria, the more plus signs the more extensively the culture grew.

WASHING EXPERIMENT.

Linen is washed for 20 minutes at 40 to 50°C. in a washing liquor, (10 g. of washing powder or soap powder to 1 l. of water). There are employed on the one hand the

washing powder or soap powder mentioned in Table 1 with an addition of 0.46% of bromamine, and on the other hand commercial washing or soap powder without additions. Pieces of equal size are then cut out from the rinsed and dried material, and are infected and treated to develop cultures. Whereas the pieces of material washed with addition of bromamine show no growth of bacteria and all the inoculated cultures die completely, a considerable growth of bacteria takes place in the cultures on the material washed with commercial washing agents without the addition of bromamine.

What I claim is:—

1. A disinfectant washing or rinsing agent containing sodium *p*-toluenesulphonbromamide and a detergent.

2. A washing or rinsing agent as claimed in Claim 1, wherein the proportion of the sodium *p*-toluenesulphonbromamide is 0.05 to 5% by weight of the final composition.

3. A washing or rinsing agent as claimed in Claim 1 or 2, containing in addition a further disinfectant in a proportion of 0.01 to 0.5% by weight of the final composition.

4. A disinfectant washing agent substantially as described in any of Examples 1 to 4.

ELKINGTON & FIFE,
Consulting Chemists and Chartered Patent Agents,
Bank Chambers, 329, High Holborn,
London, W.C.1,
Agents for the Applicants.

Leamington Spa: Printed for Her Majesty's Stationery Office, by the Courier Press.—1956.
Published at The Patent Office, 25, Southampton Buildings, London, W.C.2, from which copies may be obtained.